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TABLE 1

	Crystal Form	D10	D50	D90
1	Form-I Crystal of the Invention	5.6	12.8	25.8
2	Form-II Crystal of the Invention	5.2	11.3	22.0
3	Form-III Crystal of the Invention	4.3	8.0	14.4

D10: Cumulative undersize particle diameter at 10% of volumetric ratio [μm]D50: Cumulative undersize particle diameter at 50% of volumetric ratio [μm]D90: Cumulative undersize particle diameter at 90% of volumetric ratio [μm]

(2) Observation of the Crystals of the Invention with Electron Scanning Microscope

The crystals were observed through an electron scanning microscope (HITACHI HIGH TECHNOLOGIES TM-1000 Miniscope).

FIG. 4 shows the electron scanning micrograph of Form-I crystal of the invention. FIG. 5 shows that of Form-II crystal and FIG. 6 shows that of Form-III crystal.

From the results of (1) and (2) mentioned above, it was concluded that the particle size of Form-I crystal of the invention is larger than those of Form-II and Form-III crystals.

Test Example 2

Measurement of Residual Solvent Contained in Crystals of the Invention

The concentration of residual solvent contained in the crystals of the invention was measured by using the following measurement conditions. The result is shown in Table 2.

(Measurement Conditions)

GC Apparatus

Detector: Flame Ionization Detector

Column: Capillary Column

Column Temperature: 150° C.-230° C.

Injection Temperature: 200° C.

Detector Temperature: 300° C.

Carrier Gas: Helium

TABLE 2

	Crystal Form	Solvent	Content (ppm)
1	Form-I Crystal of the Invention	Ethanol	371
		Methyl-ethyl-ketone	82
2	Form-II Crystal of the Invention	Ethanol	2169
		Methyl-ethyl-ketone	246
3	Form-III Crystal of the Invention	Isopropyl acetate	93
		n-Butyl acetate	2781

Although each crystal Form did not contain a considerable amount of residual solvents, the amount of the residual solvents in Form-I crystal of the invention was less than those of Form-II and Form-III.

Test Example 3

Impurity-Removing Effect in Recrystallization

The effectiveness of removing impurities in the course of the recrystallization of each crystal form was measured by using the following measurement conditions (reversed-phase liquid chromatography).

(Measurement Conditions)

HPLC Apparatus

Detector: Ultraviolet Absorption Detector

Column: ODS Column

Column Temperature: 40° C.

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Mobile Phase Mixture of Water, Acetonitrile and Methanesulfonic acid

The purity (%) of each crystal of compound A was calculated by the following equation.

$$\text{Purity (\%)} = (\text{Peak area of compound A}) / (\text{Total area}) \times 100$$

The removal ratio of impurities (%) for each crystal was calculated by the following equation.

$$\text{Removal ratio of impurities (\%)} = \{[(\text{Purity of each crystal of compound A}) - (\text{Purity of crude compound A})] / [100 - (\text{Purity of crude compound A})]\} \times 100$$

The result is shown in Table 3.

TABLE 3

	Crystal Form	Purity of Compound A (%)	Ratio of Impurity Removal (%)
	Crude Material	98.04	
1	Form-I Crystal of the Invention	99.51	75
2	Form-II Crystal of the Invention	99.33	66
3	Form-III Crystal of the Invention	98.97	47

From the result shown in Table 3, the effectiveness of removing impurities for Form-I crystal of the invention was the highest compared with those for Form-II and III crystals.

Test Example 4

Investigation of Solvent for Crystallization of Compound A

Investigations of crystallization of compound A were executed according to the methods of the following (1) and (2).

(1) Crystallization solvent (see Tables 4 and 5) was added to compound A, and the mixture was stirred at 50° C. for 60 minutes. The resulting mixture was filtered. After the filtration, the isolated mother liquor was stirred at 60° C. for 30 minutes, and cooled down to 5° C. over 11 hours. After stirring at 5° C. for 72 hours, the precipitated solid was collected by filtration. The solid was dried at 20° C. under reduced pressure, whereby a solid was obtained.

Powder X-ray diffraction spectrums of the obtained crystals were measured and the form of each crystal was determined.

The results are shown in Table 4 (investigation by single solvents) and Table 5 (investigation by mixed solvents).

In the investigation by mixed solvents (Table 5), each solvent was mixed and used in an equal amount.

TABLE 4

	Crystallization Solvent	Crystal Form
1	tert-Butyl methyl ether	NA
2	Acetone	Form-II Crystal of the Invention + Form-III Crystal of the Invention
3	Chloroform	NA
4	Methanol	Form-II Crystal of the Invention + Form-III Crystal of the Invention
5	Tetrahydrofuran	Form-II Crystal of the Invention + Form-III Crystal of the Invention
6	Isopropyl ether	NA
7	2-Methyltetrahydrofuran	Form-II Crystal of the Invention + Form-III Crystal of the Invention